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FABRICATION AND TESTING OF TAA BONDED CARBON ELECTRODES IN PRIMARY FUEL CELLS, 3#

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Table of Contents

page

List of Tables

List of Figures

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FABRICATION AND TESTING OF TAA BONDED CARBON ELECTRODES

ABSTRACT

Cobalt dibenzotetraazannulene (CoTAA) has been covalently linked via an acyl ligand to carbon (Vulcan XC-72). This bonded-CoTAA has been shown, in rotating disk electrode tests, to have catalytic activity equal to platinum on a weight percent basis for the oxygen dissolution reaction at room temperature in concentrated phosphoric acid. Bonded-CoTAA has been shown to be stable in 150°C concentrated phosphoric acid. Performance of bonded-CoTAA as a cathode in a primary fuel cell will be determined in the next quarter.

1.0 Objective and Scope of Work

The objective of the program is to covalently link cobalt TAA to a carbon matrix and to use the resultant electrode material in a primary fuel cell as the active material in a wet-proofed oxygen electrode. A fuel cell using this electrode will be constructed and run. Four tasks will be carried out to obtain the data required to meet these objectives:

1. Evaluate TAA complexes to identify the desired TAA which is the most active and stable form;
2. Functionalize and covalently link the desired TAA to conductive carbon and demonstrate its stability in hot phosphoric acid;
3. Fabricate a wet-proofed oxygen-dissolution reaction electrode from the TAA-bonded carbon;
4. Test the TAA-bonded carbon electrode in a primary fuel cell at 150°C using a phosphoric acid electrolyte.

2.0 Summary of Progress to Date

2.1 Work Done During First Quarter

Twelve tetraazannulene (TAA) compounds were prepared as metallized complexes; these were purified either by recrystallization from hot cyclohexanone or sublimed under vacuum. All metallized TAAs prepared were evaluated for catalytic activity in a rotating electrode apparatus as 50% paraffin pellets containing carbon (Vulcan XC-72) with a 10% catalyst load (by weight). These pellets were inserted into the hollow-end disk electrode, subjected to five potential scans over the range of +1.1 to 0.1 V vs hydrogen and then the current on each pellet measured at +0.1 V as a function of RPM (0-10,000). The observed order of activity of the TAAs tested was:

$$\mathrm{Co} > \mathrm{Fe} > \mathrm{Ni} > \mathrm{Mn}$$

$$\mathrm{TAA} > \mathrm{NO_2\text{-}TAA} > \mathrm{Cl_2\text{-}TAA} > \mathrm{Cl\text{-}TAA}$$

CoTAA was observed to have activity similar to that of 5% platinum on carbon.

Vulcan XC-72 was bromoacylated under Friedel-Krafts conditions to provide a six-link carbon which showed by mass composition analysis 0.2 mMole bromine/g-carbon; should reaction of this bromoacylated carbon with functionalized TAA on a 1:1 stoichiometric basis be complete, a catalyst loading of approximately 7.3% (by weight CoTAA) will be achieved.

2.2 TAA Functionalization

TAA was functionalized to obtain N,N-dimethyl-aminomethyl TAA (bis-aminated TAA); this material was purified by passing through an aluminum oxide column. Attempts were made to quaternize this material before and after reaction with cobalt acetate; the results obtained showed that metallization should be done after quaternization.

2.3 TAA-Bonding

Bis-aminated TAA was reacted with bromoacylated Vulcan XC-72 under quaternization conditions. The resultant bonded-TAA was metallized with cobalt acetate in sulfolane. The observed loading of CoTAA (based on cobalt analysis) was 3.3% (by weight) on the carbon.

2.4 Activity of Bonded-TAA

The bonded-TAA carbon was examined in the rotating electrode assembly as a 50% paraffin pellet; the activity observed was similar to that of a 5% platinum on carbon pellet.

2.5 Acid-Stability of Bonded-TAA

Bonded-TAA carbon was held in conc. phosphoric acid at 150°C in quartz tubes. A rapid decrease in cobalt on the surface was observed in one day; the level of cobalt remaining stayed constant thereafter (10 days) even for an eight-fold variation in phosphoric acid volume. The level of cobalt remaining corresponds to a 1.4 % (by weight) level of loading of CoTAA on the carbon. No activity decrease was observed in the bonded-TAA carbon obtained from this test. A thirty-day stability check will be completed in October.

2.6 Electrode Fabrication

Wet-proofed electrodes of bonded carbon have been prepared (60% carbon: 40% teflon) at 320°C and 1800 psi. A gold-plated tantalum current collector (screen) was pressed into one side (320°C; 1800 psi); a sheet of 3 mil Porex was attached to the other side for wet-proofing (22°C; 1500 psi). These electrode structures have suitable physical properties to be tested in the ECO fuel cell.

2.7 The ECO Fuel Cell

A small test fuel cell was constructed using a quartz rectangular jar enclosing a pair of electrode holders and a reference electrode. The electrode holders are made from teflon blocks plumbed to permit gasing and a quartz-disk observation port to observe for electrolyte leakage through the electrode. The electrodes are held in place via a teflon O-ring against a teflon sleeve which is screwed into the teflon block. A gold wire is used to contact the electrode.

3.0 Technical Progress - 2nd Quarter

3.1 Characterization of Bonded-TAA

3.1.1 TAA Functionalization

3.1.1.1 Synthesis of Bis-N,N-dimethyl-aminomethyl TAA

One equivalent (1.37 g) of TAA (dibenzo tetraazaannulene), which had been purified by repeated crystallization in cyclohexanone followed by sublimation under vacuum, was reacted with 2.1 equivalents of dimethylamine hydrochloride and 2.1 equivalents of paraformaldehyde in 25 ml of dioxane. The mixture was held at reflux for 30 minutes, the solvent removed under vacuum and the residue redissolved in methanol. The methanolic solution was made basic with triethylamine; following removal of excess triethylamine under vacuum, the resulting solution was passed through an aluminum oxide column (Woehm basic grade I). The material obtained (1.03 g) was characterized by MS and IR to be the the desired product.

3.1.1.2 Adducts of Bis-Aminated TAA

A number of adducts of bis-N,N-dimethyl-aminomethyl TAA (bis-aminated TAA) were prepared to further develop and characterize expected products of the quaternization reaction of the bis-aminated TAA with bromoacylated carbon (Vulcan XC-72). The cobalt, the quaternized and the cobalt-quaternized adducts were prepared. During the procedure, it was observed that quaternization of the cobalt adduct with methyl bromide yielded cobalt bromide not the desired cobalt-quaternized adduct. This result showed that the bromoacylated carbon should be reacted with the bis-aminated TAA prior to metallizing with cobalt acetate.

3.1.1.3 Attempted Synthesis of Mono-Aminated TAA

A number of unsuccessful attempts were made to react CoTAA and TAA with one equivalent of N-iodomethyl-N,N,N-trimethyl ammonium iodide (a Mannich reagent); all attempts failed although the addition of two equivalents yielded the bis-aminated adduct of TAA. During these attempts it was observed that DMF (dimethyl formamide), a commonly used solvent, forms complexes with CoTAA.

Based on the negative results obtained, it is apparent that the structure of TAA is sufficiently stabilized so as not to permit single functionalization of its inner ring structure.

3.1.2 TAA-Bonding

3.1.2.1 Bonding of Bis-Aminated TAA to Bromoacylated Carbon

2 g of bromoacylated carbon (Vulcan XC-72; prepared as described in Section 3.1.2 of the Technical Progress Report No. 1, June 15, 1978) were reacted overnight with 250 mg of bis-aminated TAA in 400 ml of methanol. Two ml of methyl bromide were then added to quaternize unreacted sites on the bis-aminated TAA and the reaction mixture was allowed to stir overnight at room temperature. The TAA-carbon was harvested by centrifugation, washed with fresh methanol and resuspended in 20 ml of hot sulfolane containing 250 mg of cobalt acetate and 0.345 g of capric acid. A 0.3 g sample was removed after five minutes of reaction (to check metallization kinetics) and the rest of the mixture was stirred overnight at 100°C under argon. The bonded-CoTAA carbon was washed by centrifugation with sulfolane, methanol, water, 85% phosphoric acid and

finally water until a neutral effluent was obtained. After drying under vacuum, a 41 % total mass yield was obtained.

When this material was analyzed for cobalt, it was determined to have 3.3 and 5.3 mg cobalt/g-carbon for the five-minute and the overnight reaction products respectively. These yields are based on cobalt determination of ashed material using the procedure described in Section 3.4 of this report. Assuming all TAA on the surface is bonded and metallized, these cobalt determinations show bonding efficiencies of 27 and 42% respectively (based on bromide measured on bromoacylated Vulcan XC-72 and 1:1 reaction stoichiometry) and loading levels of catalyst of approximately 1.9 and 3.0% (by weight CoTAA) respectively on the carbon. Two further runs of making bonded-CoTAA have provided carbons with cobalt content of 6.1 and 5.7 mg/g-carbon; these results condirm the results obtained in the initial run.

Based on the three observed bonding efficiencies for the overnight reaction (42, 48 and 45%), the stoichiometry of the reaction between the bis-aminated TAA and the bromoacyl ligands on the carbon surface appears to be two: the CoTAA is apparently linked through both amino functionalities to the carbon surface.

3.1.2.2 Controls

A number of controls were prepared including unmetallized bonded-TAA, bis-quaternized TAA adsorbed to bromoacylated carbon then metallized, and bromoacylated carbon treated with cobalt acetate. These controls were all prepared using the appropriate portions of the procedure provided in the previous Section. Cobalt analysis of the controls showed the unmetallized bonded-

TAA and the metallized bromoacylated carbon (Vulcan XC-72) to have no cobalt and the adsorbed TAA to have 2.3 mg/g-carbon. The higher cobalt levels observed for the bonded-CoTAA are indicative that bonding has occurred.

3.1.3 Activity of Bonded-TAA

3.1.3.1 Activity of CoTAA Adducts

The catalytic activity of bis-aminated CoTAA and its adducts (prepared as described in Section 3.1.1) was evaluated using the standard ECO procedure in a rotating electrode apparatus. The results obtained are shown in Table 3.1; it should be noted that ECO did not attempt to develop a method of purification of bis-quaternized CoTAA (it resisted recrystallization and sublimation under vacuum).

The results obtained in Table 3.1 show that the bis-aminated CoTAA retains its catalytic activity even after heat treating (600°C) and quaternization; the level of activity is similar to that of 5% platinum on carbon.

3.1.3.2 Activity of Bonded-CoTAA

The catalytic activity of bonded-CoTAA was evaluated using the standard ECO procedure in a rotating electrode apparatus. The results obtained are shown in Table 3.2; it should be noted that the results shown in Table 3.2 are activities under oxygen diffusion limiting conditions (ie.: straight-line relationship between current observed at constant voltage and RPM$^{\frac{1}{2}}$). It was possible to obtain catalyst activity limiting conditions (ie.: region of no current change for increasing RPM) by reducing the amount of bonded-CoTAA carbon in the paraffin pellet from 50 to 17%. The activities measured under

Table 3.1

TAA Activity at $RPM^{\frac{1}{2}}$ Equal to 80 at +0.1 V vs Hydrogen using 10% by Weight Catalyst Loading

Catalyst	Activity, mA/in^2
CoTAA (600°C)	0.76
5% Pt	0.49
Bis-aminated CoTAA (crude)	0.39
Bis-aminated CoTAA (purified)	0.69
Bis-aminated CoTAA (600°C)	0.19
Bis-quaternized CoTAA (crude)	0.29

Pellets: 50% paraffin; catalyst mixed 10% by weight with bromoacylated Vulcan XC-72.

Table 3.2

Activity of Bonded-CoTAA Carbon Compared to Platinum at RPM$^{\frac{1}{2}}$ Equal to 80 at +0.1 V vs Hydrogen

Catalyst	Activity, mA/in^2
10% CoTAA (600°C) - not bonded	0.76
5% Pt on carbon	0.49
1.9% CoTAA-Carbon	0.49
3.0% CoTAA-Carbon	0.58
3.0% CoTAA-Carbon (600°C)	0.42
3.3% CoTAA-Carbon	0.54
3.6% CoTAA-Carbon	0.55

Activity measured in rotating electrode assembly with 50% carbon in paraffin pellets.

diffusion limiting conditions were observed to vary with temperature, oxygen concentration, and pellet formation technique. An attempt was made to minimize the effect of these variables in the procedures carried out.

The results provided in Table 3.2 show that bonded-CoTAA is an active catalyst for the oxygen dissolution reaction.

3.1.3.3 Comparison of Catalytic Activity of Bonded-CoTAA to Platinum

The reduction of oxygen by CoTAA and platinum can be a two or a four electron process:

$$O_2 + 2H^+ + 2e^- = H_2O_2 \qquad (1)$$

$$O_2 + 4H^+ + 4e^- = 2H_2O \qquad (2)$$

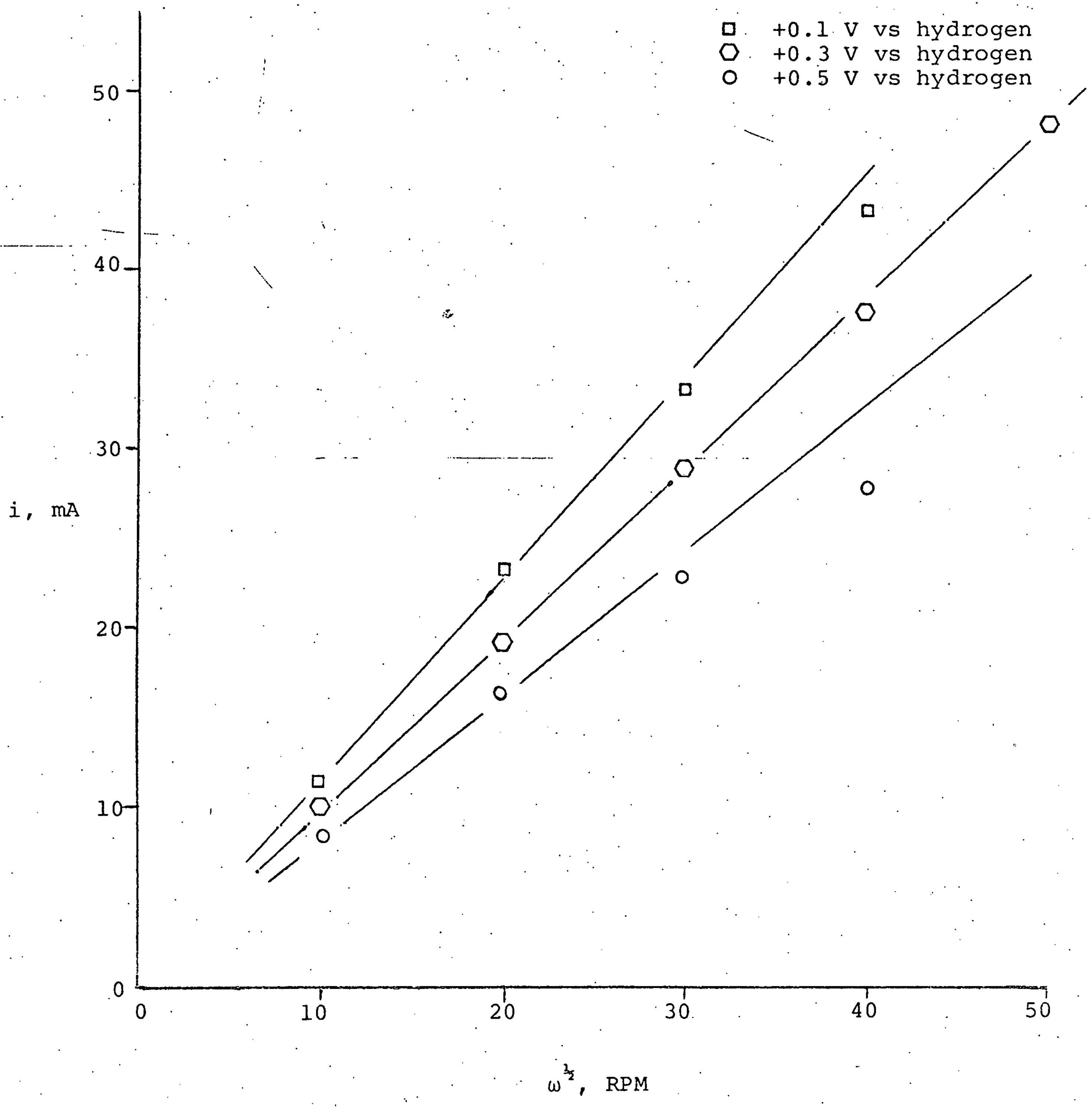
If both reactions occur simultaneously on a catalyst, the number of electrons involved in the reduction of O_2 (n) will be $2<n<4$. The use of a rotating disk electrode (RDE) allows evaluation of n as a funtion of potential. The Levich equation (Equ. 3) applies to the RDE:

$$i_L = 0.620 \; n \; F \; A \; v^{-1/6} \; D^{2/3} \; C^{\infty} \; \omega^{1/2} \qquad (3)$$

where a plot of i_L vs $\omega^{1/2}$ shoule provide a slope which is a function of n because all other terms are constant. As shown in Figure 3.1, the slope of i_L vs $\omega^{1/2}$ varies by a factor of 1.4 in going from +0.1 to +0.5 vs hydrogen with a 50% carbon pellet in paraffin with 3.6% bonded-CoTAA. This indicates that the reduction mechanism for oxygen on CoTAA (e.: Equ. 1 and 2) is potential dependent.

As follows from the theory of the rotating disk electrode (A.C. Riddiford, Advances in Electrochemistry and Electro-

Figure 3.1 i vs $\omega^{1/2}$ for 3.6% bonded-CoTAA as a Function of Potential vs Hydrogen



chemical Engineering, Vol. IV, Interscience, New York, 1966), the current through the electrode for a first order irreversible reaction, where the reverse reaction may be neglected, is given by Equation 4:

$$\frac{\pi r^2 nF}{i} = \frac{1}{k' c^{\infty}} + \frac{1}{0.62\ v^{-1/6}\ D^{2/3}\ c^{\infty}\ \omega^{1/2}} \quad (4)$$

where i is the current at constant potential and k' is the heterogeneous rate constant. A plot of $1/i$ vs $1/\omega^{1/2}$ at various potentials should give straight lines with intercepts due to the kinetic term. These plots are shown in Figure 3.2; based on the data shown in Figure 3.2, the heterogeneous rate constant for bonded-CoTAA is potential dependent with the fastest rate being observed at the lowest potential.

A comparison of the kinetics of platinum and bonded-CoTAA was carried out at +0.1 vs hydrogen on the RDE. The 5% platinum catalyst was prepared on Vulcan XC-72 as described by K.F. Blurton, et al. (Electrochemica Acta 23:183 [1978]). Pellets were made with low catalyst concentrations in paraffin so kinetic limitation conditions would be obtained. The results of this comparison are provided in Table 3.3 and Figure 3.3. These results indicate that the heterogeneous rate constant (k') for both platinum and bonded-CoTAA is large at this potential. Also the factor of two difference of slopes in Figure 3.3 indicates that oxygen is being reduced on platinum oxide to hydrogen peroxide (Equ. 1) and on bonded-CoTAA to water(Equ. 2). Below +0.15 vs hydrogen, hydrogen peroxide is known to be the principal product of the oxygen dissolution reaction on platinum (H. Behret, et al., J. Electroanal. Chem. 74:393 [1976]). A CoTAA-covered glassy carbon

Figure 3.2 1/i vs $1/\omega^{\frac{1}{2}}$ for 3.6% bonded-CoTAA as a Function of Potential vs Hydrogen

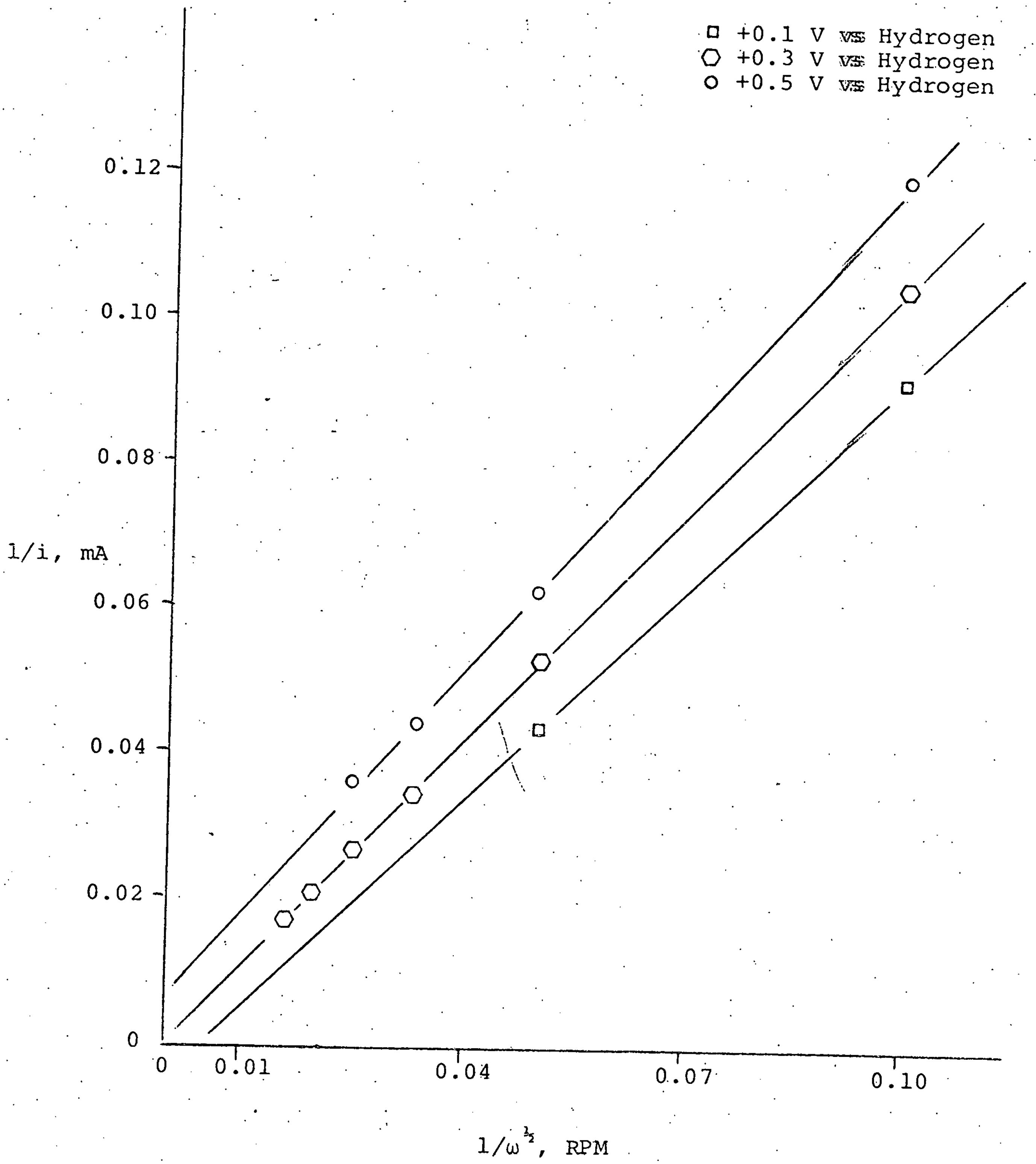


Table 3.3

Comparison of Catalytic Activity of Bonded-TAA with Platinum under Kinetic Limitation Conditions

Catalyst[1]	Loading,[2] μMoles/g	RPM of 'knee'[3]	Current at 'knee'[4] μAmps
Bonded-TAA	0	3600	2.5
5% Pt on Vulcan XC-72	43	625	4.0
Bonded-CoTAA	3.7	530	8.0

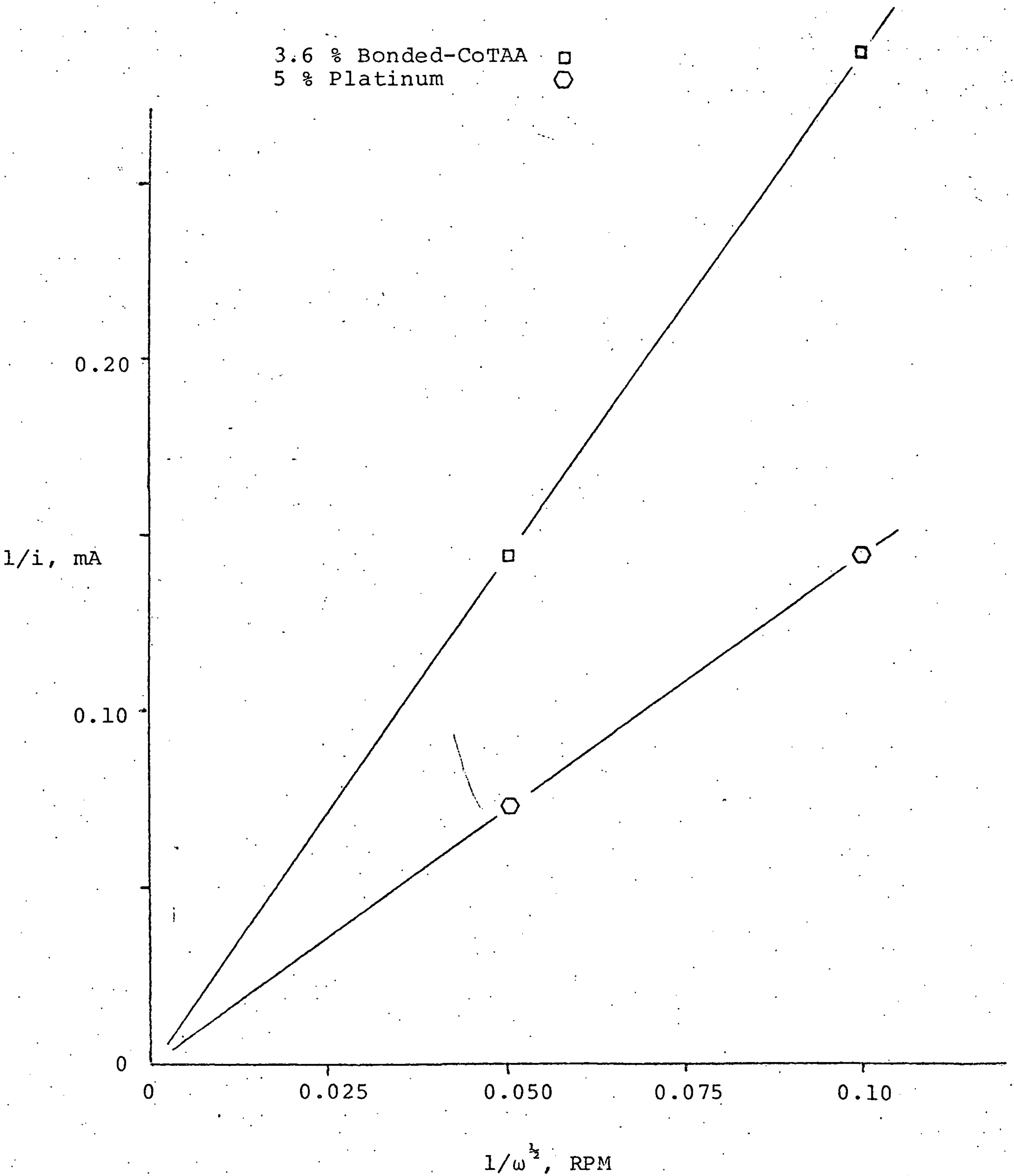
[1] Pellets were prepared by pelletizing paraffin and sample; pellets were tested in the rotating electrode assembly.

[2] Loading is the μMoles of catalyst present in the pellet per weight of the pellet (including paraffin).

[3] The 'knee' is the point on a plot of i vs $\omega^{1/2}$ where the reduction rate changes from diffusion to kinetic limitation.

[4] This current was obtained by potentiostating the RDE at +0.100 V vs hydrogen on a pellet of surface area 0.39 in^2.

Figure 3.3 $1/i$ vs $1/\omega^{1/2}$ for 3.6% Bonded-CoTAA and 5% Platinum on Vulcan XC-72



disk at +0.30 V vs hydrogen was reported to provide mainly hydrogen peroxide (H. Behret, et al., J. Electroanal. Chem. 74:393 [1976]). From Figure 3.1, it is apparent that n decreases by a factor of 1.4 going from +0.1 V to +0.5 V vs hydrogen for bonded-CoTAA. This would correspond to a 50% conversion of oxygen to hydrogen peroxide (Equ. 1) and a 50% conversion of oxygen to water (Equ. 2) at +0.5 V vs hydrogen for bonded-CoTAA.

3.1.3.4 Surface Covering by Bonded-CoTAA

A calculation was made of the extent of surface covering of bonded-CoTAA on Vulcan XC-72. This calculation was made based on known bond lengths of C-C, C-N and C-H; the calculated area of a CoTAA molecule is ∿ 164Å. For the observed level of stable bonded material (ie.: 2.4 mg cobalt/g-carbon as described in Section 3.1.4.1 and all TAA molecules linked to cobalt), the carbon surface is covered at ∿ 30 m^2/g-carbon or approximately 15% (200 m^2/g-Vulcan XC-72).

3.1.4 Acid-Stability of Bonded-CoTAA

3.1.4.1 Acid-Stability Determined by Cobalt Content

100 mg samples of bonded-CoTAA carbon were held in 2 ml of conc. phosphoric acid in quartz tubes at both room temperature and at 150°C. After one day, four days and ten days, the carbon was harvested by centrifugation, washed in methanol and dried under vacuum. The cobalt remaining on the carbon was determined by the procedure described in Section 3.4; an attempt at determining cobalt in the acid solution failed because phosphoric acid interferes with cobalt determination by atomic absorption spectrometry. Table 3.4 provides the results

Table 3.4

Cobalt Content of Bonded-CoTAA after Treatment in 150°C Concentrated Phosphoric Acid

Time, days	Cobalt, mg/g-carbon
0	6.1
1	2.4
4	2.4
10	2.4
30	n.a.

Data based on 100 mg of bonded-carbon held in 2 ml acid in quartz tubes.

obtained at 150°C; these results show that there is immediate loss of cobalt in the first day followed by no further loss. This observation was duplicated in the same test run at room temperature. A further experiment was run in which the volume of phosphoric acid was varied (0.5 to 4 ml); as shown in Table 3.5, the level of cobalt remained at 2.4 mg cobalt/g-carbon independent of acid volume. Heat-treatment (600°C) of bonded-TAA did not significantly affect the level of cobalt remaining.

Two controls were run: bromoacylated Vulcan XC-72 to which was added 6 mg cobalt (as cobalt acetate)/ g-carbon; bromoacylated Vulcan XC-72 which had bis-quaternized TAA adsorbed and then metallized as described in Section 3.1.2.2. When these two controls were held in 150°C phosphoric acid, residual cobalt measured was 0.01 and 1.0 mg cobalt/g-carbon respectively after ten days.

These results on cobalt content show that the bonded-CoTAA is stable in 150°C phosphoric acid: the level of stable bonded-CoTAA is equal to a catalyst loading of 1.4 % (as CoTAA) on carbon. A 30-day stability check will be completed during October, 1978.

3.1.4.2 Acid-Stability Determined by Activity

The carbons obtained from the tests described in the previous Section were tested in the rotating electrode assembly by the usual procedure as 50% carbon in paraffin pellets. The results obtained are shown in Table 3.6; these results show that there was no significant activity change under oxygen diffusion limiting conditions for the bonded-CoTAA after treatment for 10 days in 150°C conc. phosphoric acid.

Table 3.5

Cobalt Content of Bonded-CoTAA after Treatment for One Day in Varying Amounts of 150°C Conc. Phosphoric Acid

Volume Acid, ml	Cobalt, mg/g-carbon
0.5	2.4
2.0	2.4
4.0	2.4

Data based on 100 mg of bonded-carbon held in quartz tubes.

Table 3.6

Catalytic Activity of Bonded-CoTAA after Treatment in 150°C Conc. Phosphoric Acid

Catalyst	Activity, mA/in^2	
Days:	0	10
3.6% CoTAA-carbon	0.55	0.41
3.6% CoTAA-carbon (600°C)	0.54	0.56

Activity measured as 50% carbon in paraffin pellets in a rotating electrode assembly.

3.1.5 Summary

Bonded-CoTAA has been made and evaluated at 150°C in conc. phosphoric acid; it has been shown to be of similar activity to platinum (by weight) as a oxygen dissolution reaction catalyst and to be stable in 150°C conc. phosphoric acid.

3.2 Electrode Fabrication

3.2.1 Electrode Press

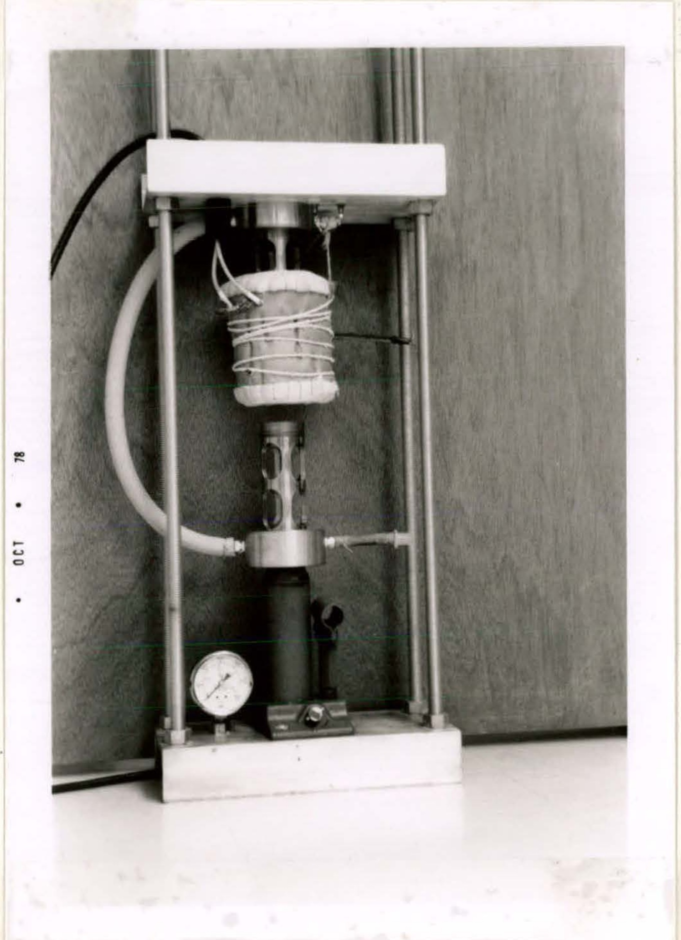
An electrode press (Figure 3.4) was designed and fabricated; this press permits forming of 1½ in. dia. electrodes at elevated, but controlled, temperatures and pressures.

3.2.2 Electrode Fabrication

Test electrodes have been fabricated using a mixture of 60% carbon (1.5 g) - 40% teflon. This mixture was prepared by mixing the desired amount of carbon into the teflon suspended in Freon 133 (100 ml) in a Waring blender. The teflon was first washed with water/methanol and then dried under vacuum. After the Freon 133 was driven off at room temperature, the mixture was placed in the press and formed into an electrode (1800 psi; 320°C). The resulting electrode was conductive and physically stable.

A metal grid was pressed into one side of the electrode (1800 psi; 320°C). This grid was made of gold-plated tantalum where the gold plating was achieved by cleaning the tantalum in hydrofluoric acid followed by plating to a soft gold surface using an Aurotemp 20 solution from Technic, Inc. Control grids left in 150°C conc. phosphoric acid showed no degradation

Figure 3.4 Electrode Press



• OCT • 78

after 10 days.

The electrode was 'wet-proofed' by pressing a sheet of 3 mil Porex (a porous teflon) on the surface at 22°C and 1500 psi.

3.3 The ECO Fuel Cell

3.3.1 Cell Fabrication

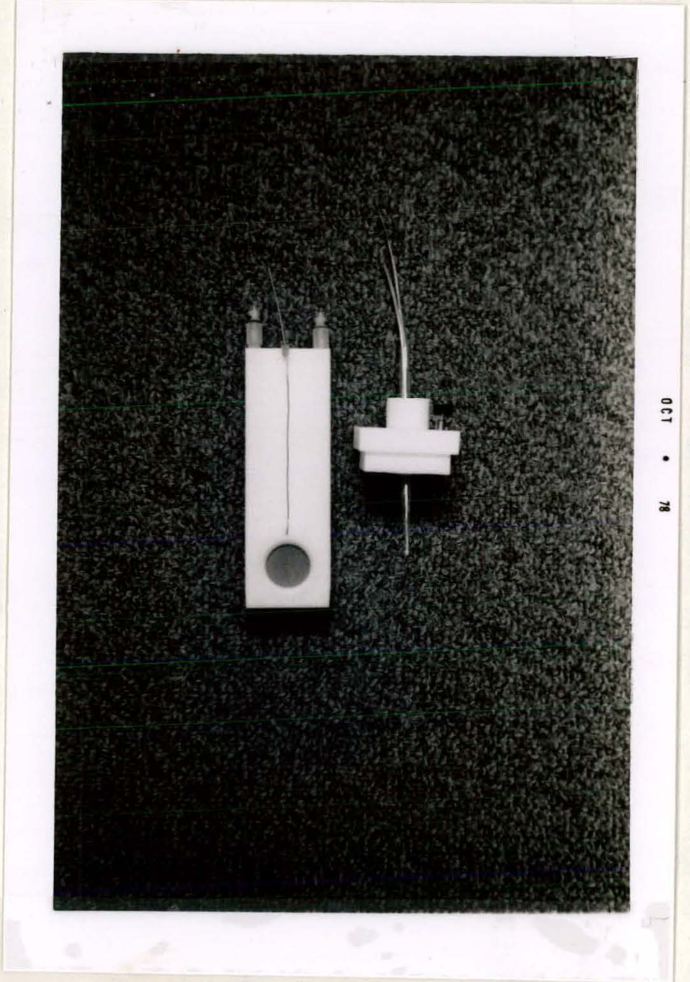
A cell has been constructed which consists of a 2" x 3" rectangular quartz jar encasing a pair of electrode holders and a reference electrode. Shown in Figure 3.5 is an electrode holder and a hydrogen reference electrode.

The electrode holder consists of a block of teflon plumbed to permit gas flow through the block to the electrode. The electrode is held via an O-ring and a teflon sleeve with the gold-plated grid tight against a gold wire current-collector. A clean quartz disk is held against the sleeve by a second sleeve which is screwed into the teflon block: the electrode is held against electrolyte leakage into the gas chamber by tightening the second sleeve. The gas chamber and the electrode can be observed through the quartz disk to verify that it has not become flooded.

3.4 Cobalt Determination Procedure

A general procedure for determination of cobalt in carbon (or carbon-paraffin) was developed. The general procedure is as follows: 100 mg of dry, washed carbon containing cobalt was placed in a ceramic boat and ashed at 800°C in air. The ash was dissolved in conc. HCl by heating at reflux for ten minutes. The resulting solution was diluted to a known volume (2 or 10 ml) and analyzed by atomic absorption

Figure 3.5 Electrode Holder and Reference Electrode



spectrometry for cobalt. Direct measurements on the order of 100 to 130 ppm cobalt were obtained which are at a suitable level for accuracy and sensitivity.

Five controls were run(two with paraffin; three without) with bromoacylated Vulcan XC-72 mixed with 10% pure CoTAA; Table 3.7 shows the results. The percent recovery obtained is independent of the presence of paraffin and is approximately 70%; all results provided in this report are actual results adjusted by this recovery factor.

Table 3.7 Cobalt Determined by Ashing Bromoacylated Vulcan XC-72 with 10% CoTAA

Trial #	Co, ppm	% Recovery
1	110	65
2	123	73
3	118	70
4 (with paraffin)	120	71
5 (with paraffin)	115	68